5120 – Advanced Metallics Branch – Highlights – FY00

Successful validation on production-scale material of the improved mechanical properties for the new powder metallurgy large engine disk alloy ME3 and regional engine disk alloy Alloy 10, which are slated to be the first significant new disk alloys introduced by GE, P&W, and Honeywell in 15 years. **(Gabb, Gayda, Garg, Ellis, O'Connor, Telesman, Kantzos)**

Chemistry modifications to the powder metallurgy superalloy Alloy 10 demonstrated further improvements to mechanical properties and processability. **(Gayda, Telesman, Kantzos)**

Casting technology for producing superalloy Lattice Block {a bridge truss-like structure} materials has been developed in conjunction with JAM Corp. This is the first step in an effort to produce lightweight, very strong structures for use at elevated temperatures. **[Hebsur, Bartolatta, Whittenberger, Hopkins, Krause].**

Ballistic impact tests were performed on over 20 candidate fan containment **materials**. At low areal weights (<1.5 lb/ft^2) Glare panels, which are laminated composites composed of Al-alloy sheet/glass fiber prepregs, performed best. At higher areal weights (>2 lb/ft^2) conventional materials such as Inco 625 and 304 SS performed best. Depending on the test material, sample thickness, and penetrator velocity, there is a change in impact failure mechanism that accounts for a change in ranking of material performance over different areal weight ranges (because of changes in sample thickness). **(Noebe, Hebsur, Revilock)**

The effect of ballistic impact damage on fatigue of TiAl was extended to a high strength, lower ductility alloy known as ABB-2. It was found that the impact and fatigue resistance of both ABB-2 and Ti-48Al-2Nb-2Cr were essentially identical. Remnant fatigue strength can be accurately predicted based on the surface crack lengths resulting from the ballistic impacts. **(Draper, Lerch, Pereira, Nathal, Juhas, Veverka)**

A study of cryomilled NiAl mixed with 10 vol % Cr improved room temperature toughness by a factor of two {from 5 to 11 MPa m½} while the elevated temperature strength was only slightly decreased. **[Whittenberger, Aikin, Salem]**

A dual alloy titanium impeller with enhanced speed and temperature capability was developed by a NASA/Honeywell/GE team. The impeller employed a high-strength titanium alloy (Ti-6246) in the bore that was bonded to a high temperature, creep resistant titanium alloy (Ti-834) in the "airfoil" section. Elevated temperature spin testing showed the impeller could withstand speeds in excess of 130% of design requirements without any distress at the bond line. **(Gayda)**

Tensile, fatigue and creep rupture testing of thin gage TiAl alloys made by new lower cost processing methods has shown that all the mechanical properties are reproducible and comparable to those measured on cast versions. The combination of lower sheet processing cost with good strength makes this material attractive for aerospace applications. **(Whittenberger, Locci, Draper, Bowman)**

In a cooperative program with MSFC, the advanced copper alloy GRCop-84 was successfully tested under realistic rocket engine conditions. Both uncoated and NiCrAlY-coated spool pieces were hot fire tested for 17 cycles totaling approximately 500 seconds, at oxygen/fuel ratios up to 7.0, the equivalent of a 6,000°F flame. The testing demonstrated that VPS is a viable method for producing combustion chamber liners. It also demonstrated that functional gradient coatings that are an integral part of the combustion chamber liner can withstand rocket engine conditions. **(Ellis)**

Provided answers to key questions regarding the feasibility of IN-718 sheet intended for the heater head of a Stirling engine as a power source for deep space missions. Based on in-house creep and microstructural analysis, in addition to literature data, an optimum grain size and processing scheme was specified and verified to meet the 100,000-hour life requirement. These specifications were used in the procurement of the IN718 bar stock, which will be used in the production of the actual flight hardware. **(Bowman)**

2000 Highlights - Ceramics Branch (5130)

Multilayer Thermal Radiation Reflector Coatings: Multilayers of aluminum oxide and yttria stabilized zirconia are being investigated for thermal radiation reflector coatings on oxide ceramic matrix composites. Modeling of alternating layers of various thickness has indicated the coatings are capable of reflecting 50% or more of the thermal energy away from the hot surface thus effectively lowering the CMC temperature and reducing thermal gradients. Current modeling efforts are aimed at optimizing the layered structure and increasing reflectivity of the coating. **(Charles Spuckler (5820) and Martha Jaskowiak)**

Whisker Reinforced Oxide Matrix Composites: Mullite whisker preforms have been fabricated for use as reinforcement in oxide matrix composites. The whisker processing method offers the capability of producing preforms in near net shape dimensions. The preforms can be infiltrated with a matrix without the need for hot pressing as a final densification process. As-produced preforms possess a rigid interlocking network of randomly oriented whiskers that is retained in the final part. Recent work was aimed at controlling the whisker morphology and aspect ratio. Processing conditions were identified which led to increased aspect ratios with a more uniform distribution of whiskers throughout the performs. **(Martha Jaskowiak and John Setlock)**

Affordable Silicon Carbide Ceramics Fabrication Technology Transferred to Bechtel Bettis, Inc.: Advanced silicon carbide-based ceramics and fiber-reinforced composites are being actively considered for a number of components in the nuclear industry. The affordable silicon carbide ceramics fabrication technology, which was developed in-house, was successfully transferred to Bechtel Bettis, Inc., Bettis Atomic Power Laboratory, West Mifflin, PA under a reimbursable project for the fabrication of ceramic components for nuclear applications. **(M. (Jay) Singh and Rich Dacek)**

Al-B-N Coating Possible Promising Alternative to BN: Work with HyperTherm, Inc. under SBIR funding to develop interphase for longer CMC life in oxidizing environments has produced an Al-B-N coating that shows excellent mechanical performance in minicomposite testing. CMC panels have been fabricated and are being evaluated. **(Fran Hurwitz)**

CMC Tail Cone: GRC, in partnership with Allison, Northrop Grumman and the Air Force, contributed to the development of processing parameters for a Blackglas™ CMC tail cone that has been successfully engine tested on an Allison 2100. The material also is of interest as an exhaust tailcone on the Allison 3007 and for the Global Hawk Tier II aircraft. **(5130/ Fran Hurwitz, and 5160/Mary Ann Meador)**

Micromechanical and Dielectric Property Measurement Capability: Aluminum nitride has been shown to have great potential as a high temperature electronic packaging material for advanced high component density electronic devices as well as piezoelectric capabilities. In house capabilities to nondestructively characterize mechanical damping and elastic modulus have been expanded to include electrical properties. Results at temperature up to 1000^{o}C indicate intrinsic damping in aluminum nitride is too weak to suppress mechanical vibrations. Also, alternating stress and electrical fields induce dipole reorientation of vacancy defects thus providing a quantitative method for determining product purity. **(Jon Goldsby)**

NASA GRC Ceramic Matrix Composites (CMC) Development Team Contributes to Success of 2400^{o}F CMC Development: The GRC CMC Development Team has been providing key innovations in fiber pretreatment to enable Honeywell Advanced Composites to develop an advanced SiC/SiC composite that can operate at 2400^{o}F in engine hot sections. Honeywell is funded by an Air Force contract. The experience gained in this collaboration will facilitate the attainment of UEET goals for a durable CMC operating at 2700^{o}F surface temperature and 2400^{o}F material temperature for combustor liner and vane applications. NASA approaches for 2400^{o}F SiC/SiC composites may also be implemented by the Air Force in IHPTET and related programs. **(Jim DiCarlo)**

Mechanistic Model for Intermediate Temperature Stress-Rupture of Ceramic Matrix Composites (CMC): A model was developed to predict the time to failure of woven SiC/SiC composites subjected to stress-rupture in air at 815^{o}C. The model is based on the physical mechanisms that lead to failure for composites with through thickness matrix cracks. For this case, predictions were in excellent agreement with experimental data. For less than fully cracked matrix conditions the model underestimated life. The current work is aimed at applying this model to stress-relaxation conditions, and expanding the model for microcrack growth to make the model more useful at regions of high stress-concentration such as notches and holes. **(Greg Morscher)**

Doped boron nitride interphase coatings for improved oxidation resistance in ceramic matrix composite (CMC) components: Studies performed under the Enabling Propulsion Materials program demonstrated that boron nitride (BN) coatings provided appropriate composite mechanical behavior in CMCs, but were susceptible to oxidation; however, doping of boron nitride with low levels of silicon improved oxidation resistance. In preliminary work under the Propulsion Systems Research and Technology Program, a method has been demonstrated on fiber tows for achieving a BN coating with10-12 atomic percent of uniformly distributed silicon (Si). Work is continuing to apply the Si doped coating to fabric, and to minimize oxygen incorporation in the coating process by moving from a continuous tow coater to a batch reactor. **(Fran Hurwitz)**

Improved Constituent Materials and Processes for SiC/SiC Hot-Section Components: The successful application of ceramic matrix composites as hot-section components in advanced engines will require the development of CMC material systems that are capable of displaying not only long-term structural performance above 2200^{o}F, but also high thermal conductivity and the ability to resist engine environmental attack at all temperatures. Initial progress in identifying constituent materials and processes to achieve these goals was made under the former NASA EPM Program using Sylramic SiC fibers, 0/90 2D-woven fabrics, BN interphases, and CVI SiC plus melt-infiltrated (MI) Si-SiC matrices. Under the new NASA UEET Program, continued progress in constituent optimization for this SiC/SiC system has been made. This includes the development of specific thermal treatments at NASA-Glenn that significantly improve the creep and environmental resistance of the Sylramic fiber (in-situ BN coating formation) as well as the thermal conductivity and creep resistance of the CVI SiC constituent of the matrix. Experimental and theoretical studies have established improved guidelines for continued optimization of constituent materials and processes for SiC/SiC component subelements such as panels, cylinders, and tubes. **(Jim DiCarlo, Ram Bhatt, Greg Morscher, Hee Mann Yun (5120), and Linus Thomas-Ogbuji (5160))**

Characterization of heat treated SiC fiber preforms: Intrinsic strength and microstructural stability of BN/SiC coated Hi-Nicalon, Hi-Nicalon-S, and Sylramic fiber preforms have been studied in the temperature range between 25 and 1800^{0}C in argon environment for up to 100 hr to determine the most stable preforms for fabrication of SiC- and silicide-based composites stable to 1315^{0}C(2400^{0}F), as well as to determine the influence of preform heat treatment on composite properties including improved thermal conductivity. Among the three preforms studied, the Sylramic fiber preforms are stable to 1700^0C. **(Ram Bhatt)**

National Space and Missile Materials Symposium: GRC played a major role in organizing and carrying out the National Space and Missile Materials Symposium, February 27-March 2, 2000 in San Diego, CA. Involvement included arranging for partial financial support as noted below, Steering Committee membership, administrative support at the conference, and setting up and staffing a Materials and Structures exhibit of GRC capabilities and accomplishments. The Space Directorate, Space Transportation Projects Office, and the Propulsion and Power Technology Base R & T Program Office, and the Aeronautics Directorate, High Speed Research Office, provided partial financial support for the conference. **(Doug Kiser)**

Ceramic Matrix Composite (CMC) Tests Successful: The Rockwell Science Center has been developing open woven structure architecture ceramic matrix composites for cooled nozzle ramp and other propulsion applications under an Air Force Research Laboratory contract under the Integrated High Payoff Rocket Propulsion Technology Program (IHPRPT). The GRC Ceramics Branch was part of the contract team with the primary task being to test the thermal performance of the panels under a variety of aero thermal conditions. Each panel was to subject to eight conditions where the chamber pressure and the mixture ratio of the H_2/O_2 thruster were the primary variables. Each test condition increased in severity, culminating with the maximum output of the thruster at a chamber pressure of 500psi and an oxidizer-to-fuel ratio of 7.0. Both the polymer-infiltration-pyrolysis and melt infiltration densified panels survived the maximum fury of the thruster without any visible damage. **(Andy Eckel)**

Ceramic Propellant Designed, Fabricated, and Demonstrated: Ceramic propellant injectors offer the potential for significant weight reductions and can enable new designs for a broad range of combustion devices. A propellant injector faceplate has been fabricated using laminated object manufacturing and standard ceramic processing by CWRU and NASA GRC Ceramics Branch personnel. Preliminary test results have proven successful. Design and fabrication processing optimization continues **(Andy Eckel and Jim Cawley(CWRU)).**

C/SiC Database and Summarization of C/SiC Literature: C/SiC is an important ceramic matrix composite (CMC) material for space transportation applications. A database of C/SiC literature has been prepared at GRC that currently includes over 170 documents, with these references categorized by subject and author. The intent of this effort is to maintain a database so that information on current C/SiC research is readily available and the information has been provided to other researchers upon request. The properties obtained using different processing approaches has been summarized and presented at a technical meeting (Cocoa Beach, Restricted session 2000) and documented in the proceedings. **(Paula Heimann)**

Oxidation Model for C/SiC Composites: A finite difference model that uses physics based theories for the binary diffusion of gasses and the reaction kinetics was further advanced to consider a cross-section that more closely matches that of experimental test coupons. The model is able to track carbon consumption and oxygen concentrations throughout a cracked, fiber-bridged, cross-section over time. The model also keeps track of the amount of carbon consumed at the perimeter of the fiber tows in addition the amount of bulk carbon that has reacted. The oxidation trends, the amount of carbon reacted over time, and the predicted oxidation kinetics compared very well to the experimental results and microstructural analyes from stressed oxidation tests conducted at temperatures of 800°C and 1200°C at a stress of 20 ksi in an air environment. **(Mike Halbig)**

Light-Weight Gas Generator Combustor: The design phase for the light-weight gas generator is complete. Finite element analysis of the design has been conducted and modifications implemented where warranted. The design consists of concentric C/SiC braided cylinders densified using the melt-infiltration process. Braid angles and tows were selected to provide optimum fabrication results while maintaining structural integrity. Three sets of combustor cylinders are currently being fabricated; one reserved for permeability testing and two for hot-fire testing in March 2001. Once testing is complete a Technology Insertion Roadmap document will be generated. **(Jerry Lang, Andy Eckel)**

2000 Polymers Branch Major Accomplishments

- Efforts to develop non-toxic replacements for PMR-15 have continued over the past year. Development of a non-toxic replacement of PMR-15 could save the aircraft engine industry alone millions of dollars per year in handling and disposal costs associated with the use of PMR-15. The HOTPC, UEET and SBIR programs have supported research in this area.

  - In a collaboration between GRC, St. Norbert College, and the University of Akron an extensive evaluation of fluorinated diamines has been concluded. Two potentially viable candidate replacements for MDA in PMR polyimides were identified **(P. Delvigs, D. Hardy-Green (U of Akron), D. Klopotek (St. Norbert College))**

  - Triton Systems (SBIR Phase II) has developed a new approach for the preparation of BAX diamine, a leading replacement for MDA in PMR polyimides. This new approach reduces the number of synthetic steps from three to two and should result in a substantial reduction in manufacturing costs. The cost of the BAX diamine has been a major barrier to the commercial success of this potential PMR-15 replacement material.

  - Further development of BIP and BIM resins has continued at GRC and Maverick (SBIR Phase II). These non-toxic polyimides have been modified to reduce their melt viscosity and enable their processing by Resin Transfer Molding **(R. Vannucci, R. Gray (Maverick)).**

- Continued to work on reducing the cost of composite components by attacking manufacturing costs. This work has focused on the development of high temperature polymers with melt viscosities low enough for processing by Resin Transfer Molding (RTM) or Resin Film Infusion (RFI) and on the use of radiation curing (electron beams or ultraviolet light), rather than heat, to cure high temperature polymers . Use of RTM or RFI processing can reduce component fabrication costs by as much as 50% over traditional autoclave or compression molding. Use of radiation curing can lead to substantial reductions (up to 30%) in tooling and manufacturing costs. Both technologies are required for the fabrication of large structures, such as cryotanks for Reusable Launch Vehicles. Work in these areas has been supported by the HOTPC and Space Transportation (Spaceliner 100) programs.

  - As part of a collaborative program with Boeing and LaRC, two GRC developed phenylethynyl terminated polyimide composites, HFPE and HFBZ, have been successfully fabricated by RFI at Boeing. These composites exhibited good compressive strength and thermal-oxidative stability at 288 and 316ºC. and are currently under evaluation for use in RLV airframe and propulsion applications **(K. Chuang, D. Hardy-Green (Akron), C. Arendt (Boeing)).**

  - In a collaboration with the U. of Akron and Prairie View A&M University, new branched polyimide formulations were developed with melt viscosities low enough for RTM and RFI processing. These new polymers had Tg's as high as 320ºC, which suggests that they might be suitable for long-term use at 288ºC. Composite panels have also been prepared from these resins at GRC using a crude RFI technique. **(T. Oliver (PVAMU), M.Meador, C Gariepy (Akron), B. Nguyen (Akron)).**

  - Continued efforts to develop radiation curable high temperature polymers as a collaboration between GRC, the University of Delaware, Kent State University and

Adherent Technologies. This effort is supported by the newly constructed Northeast Ohio Regional Beam Alliance facility at Kent State University. A GRC in-house capability has been established to utilize ultraviolet light curing in the preparation of thin films. UV curable polyimides developed under this program were one of four materials technologies featured by NASA at SAMPE2000. **(C. Malow (SHARP), M. Meador)**

- Durability of polymeric materials (resins, adhesives, and composites) is being addressed under several programs in an effort to improve component reliability and increase safety. Research in this area is supported under the Ultrasafe, HOTPC, and Space Transportation programs.

  - Re-developed an in-house capability at GRC in the processing and testing of adhesives **(D. Papadopoulos, K. Chuang).**

  - Conducted evaluation of composites for missile applications under a collaboration with the Naval Air Weapon Center and Boeing. Stitched composites prepared from GRC's DMBz-15 polyimide were the only laminates tested that survived a simulated mission profile including 10 to 15 minute exposures at 425 to 537ºC **(K. Chuang, K. Bowles, D. Papadopoulos).**

  - Completed specimen design, fabrication, high strain rate testing, and constitutive modeling of carbon/epoxy composites. Results were presented at the ASME International Mechanical Engineering Congress and Exposition, Orlando, FL, November 5-10, 2000. The data is currently being used in explicit finite element codes to model the impact dynamics of composite fan containment structures. **(G. Roberts, R. Goldberg (5920), M. Pereira (5930)).**

  - Completed ballistic impact tests on four types of carbon/epoxy composite plates with 3D woven fiber architecture. The effectiveness of the transverse fiber reinforcement in reducing delamination was demonstrated, however high material and fabrication cost is an issue for commercial use. The 3D composites are being evaluated for use in composite containment structures**.(G. Roberts)**

  - Designed and built a new high temperature erosion test lab under a HOT PC funded collaboration between 5150 and 5160. This lab will be used to test erosion coatings developed for high temperature polymer composites. The erosion lab, which complies with ASTM specifications, has the capability of thermal cycling specimens during erosion testing. This unique capability will be a more stringent test of coating durability than standard isothermal testing. **(J. Sutter, M. Cuy (Dynacs, K. Miyoshi (5160))**

  - Initiated two new programs under the HOTPC program to develop durable polymer matrix composite components for aerospace applications. Allison Advanced Development Corporation (AADC)/Rolls Royce America and a GRC team from 5100/5900 will develop coatings to improve the erosion resistance of high temperature polymer composites for commercial and military advanced gas turbine engines. Boeing Rocketdyne and a GRC team from 5100/5900 will investigate the use of lightweight polymer composites in reusable launch vehicle propulsion applications. Engine tests in both projects are scheduled to test the durability of materials developed at NASA GRC **(J. Sutter).**

- Determined the effects of long-term aging at elevated temperatures on the compression properties of GRC developed advanced composites. Relationships between degradation mechanisms and changes in compression properties have been identified that will enable the development of models that can reliably predict the long-term durability of composites under these simulated use conditions. **(K. Bowles)**

- Designed and performed thermal analysis that helped understand the degradation of HST thermal insulation materials **(J. Sutter, D Scheiman (Dynacs))**

- Identified organic/polymeric materials and proposed test matrix for space-based Stirling engine applications for the Europa Deep Space Probe **(J. Sutter)**

- Initiated new efforts in polymer based nanotechnology and biotechnology with funding from the Zero CO2 Emissions Technology, PERS, Nanotechnology and RAC programs.

  - Developed new polyimide/clay nanocomposites for advanced aerospace applications. New thermoplastic polyimide/clay nanocomposites are being evaluated for use in cryotanks. Moisture absorption in these composites is reduced by 10 to 35% over the base resin. New thermoset based nanocomposites have been prepared from PMR-BAX, which show a 16% increase in modulus over the base resin and 20% decrease in weight loss after aging for 300 hours at 288ºC. **(S. Campbell)**

  - Coauthored winning proposal to the DoD (with 5900 and the Cleveland Clinic) to develop analytical models for human tissue to be used in the development of "virtual surgery" capabilities for use in battlefield applications and for surgeon training**. (A. Freed, D. Hopkins (5900)).**

  - Initiated new program with 5160 in the use of molecularly engineered materials (rod-coil polymers, discotic liquid crystals) as improved polymer electrolytes for lithium/polymer batteries for satellite and space transportation applications. **(M.Meador, M.A. Meador (5160))**

  - Initiated new programs in functionalized carbon nanotubes (RAC), fuzzy fibers (joint with 5120 and NCMR, RAC), improved polymeric materials for fuel cells (joint with 5520 and 5160, RAC), and supramolecular materials (Nanotechnology).

- Developed new capabilities within the Branch to support current and future GRC programs.

  - Expanded thermal analysis capability through the procurement and installation of a mass spectrometer detector for current TGA/FTIR system. **(D. Scheiman (Dynacs)).**

  - Developed new NMR imaging capability for use in determining Li ion mobilities in polymer electrolytes developed under the PERS program **(C. Johnston)**

  - Developed new biaxial test capability for use with resins and elastomers. This test rig will be used to validate analytical models for viscoelastic and viscoplastic deformation of polymers under Ultrasafe **(A. Freed, D. Gerrard (Akron))**

- Several Polymers Branch personnel have been recognized over the past year for their technical accomplishments.

- **W. Alston (Army), D. Scheiman (Dynacs) and G. Sivko (OAI)** were the recipients of a 2000 R&D-100 Award for their invention, "Extended PMR Shelf Life Technology". This modification to PMR resins and prepreg significantly extends their shelf lives and eliminates the need for low temperature storage and shipping of these materials. Use of PMR Extended Shelf Life Technology could lead to a substantial reduction in component costs by reducing shipping and handling requirements and scrap rates.

- **A. Freed** was named a Fellow of the ASME

- **K. Chuang** received the NASA Exceptional Achievement Medal.

- **B. Nguyen** and **C. Gariepy**, two NASA GSRP students from U of Akron, successfully defended their thesis research and received the PhD in Polymer Science. Both students performed their thesis research at GRC.

SPECIAL THANKS TO OUR TID AND AKIMA SUPPORT STAFF AND TO SANDY CLAY FOR CONTRIBUTING TO THESE ACCOMPLISHMENTS!!

Environmental Durability Technical Accomplishments 2000

Aerospace Propulsion and Power Program:

- Acoustic emission and image analysis of moisture-assisted spallation of alumina scales on pre-oxidized Rene'N5 indicate a possible synergy between sulfur interfacial segregation and moisture-induced stress corrosion at the interface. (Smialek, Morscher)

- Completed initial laser pulse enhanced planar and tubular test configurations to achieve laboratory high heat-flux, high cycle thermal fatigue tests for combustor materials under simulated pulse detonation engine conditions. Haynes 188 specimens, exposed under enhanced laser pulse conditions at ~1600-1650°F. After 350 hrs (700 30-min cycles) micro cracking and spallation was observed. (Fox, Zhu, Miller, Kalluri)

- An impact gun located in the Mach 0.3 Burner Rig Facility to begin foreign object damage testing of coated, cooled Si_3N_4. (Fox, Cuy).

- Analysis of Pt-modified aluminide coatings containing Hf additions showed evidence that the Hf increased adherence of the alumina scale which grows between the bond coat and top coat, which directly relates to longer TBC lives (3-4X). (Nesbitt)

- Reconfiguration of a Mach 0.3 Burner Rig for room temperature to 650oF erosion testing of PMC coatings was completed and required performance metrics were achieved. Introduced non-contact surface profiling capability. (Miyoshi, Cuy, Sutter, Miller, Meador, Kostyak)

- Expanded investment in computational thermodynamics via development of expertise in the use/application of ThermoCalc, purchase/modification and continued development of key material databases. (Jacobson, Copeland, Opila)

- Cyclic oxidation studies (1150^{o}C for 1000 1-hr cycles) of Rene' N5 with varying yttrium levels indicate that the critical Y-doping level required for good scale adhesion is 10 ppma, suggesting that the critical yttrium-to-sulfur ratio is about one-to-one on an atomic basis. (Smialek)

- Baseline experiments on cast gamma-TiAl in contact with Inconel 718 identified the effects of operating parameters of fretting (amplitude, frequency, and temperature) on the fatigue life of TiAl, at RT and 550°C. Effects of fretting on fatigue failure stresses are under study. (Miyoshi, Lerch)

- Established the oxidative superiority of NASA's alloy GRCop-84 (Cu--8Cr-4Nb) over the state-of-the-art alloy NARloy-Z (Cu-3%Ag-0.5%Zr) in the intermediate temperature range, below 700oC. This finding is a significant boost to the chances of GRCop-84 application as thrust-cell liner and vane in future-generation reusable launch vehicles. (Thomas-Ogbuji)

- A new thermo-oxidative bond coat was developed and evaluated, with an erosion resistant topcoat, on a PMC substrate for gas turbine applications up to 280oC. The bond coat, composed of Zn with up to 5% polyimide, was necessary due to substrate-top coat incompatibility and improved post-aging tensile properties. (MAB Meador, Sutter, G. Leissler, Shin, McCorkle)

- A \$6.5M, five-year research partnership with Rolls-Royce Allison, Howmet, Cannon-Muskegon and the Purdue Research Foundation, has been successfully completed. The research explored development of advanced superalloy materials, evaluation of advanced casting techniques, and wheel designs to produce integrally-bladed disks, with a mechanically optimized grain morphology with elongated grains in the airfoil and fine grains in the hub. Engine tests demonstrated 2X component life and an affordability study indicated a 15% increase in customer value. (Nesbitt)

- Completed design/development/implementation of specialized user-system interfaces, data acquisition/analysis, instrumentation control and presentation/reporting software systems in the Characterization, Mass Spectrometry and TGA Laboratories. (Auping)

- A series of new candidate polymer electrolytes for lithium polymer batteries were synthesized. The rod-coil polymers form rubbery films with low Tg (-50 oC) by solution casting polyamic acids and heating to 200 oC to imidize. (Meador, Bennett)

- A possible replacement for the norbornenyl end cap (7-hydroxynorbornene-2,3-dicarboxylic acid) has been identified. The new end cap's hydroxy-bearing carbon is more easily oxidized to carbonyl on aging in the polymer, and therefore more highly favors bridge degradation, which can lead to lower weight loss on aging. (Meador, Frimer)

Ultra Efficient Engine Technology Program:

- Upper temperature limit of EPM environmental barrier coatings (EBC) was estimated to be 1400 ^{o} C, based on water vapor stability, environmental and chemical durability, phase stability and thermal conductivity. Glass formation, due to BSAS/silica reaction, and BSAS vaporization, by water vapor reaction, were the key limiting factors. (Lee, Fox, Eldridge, Zhu, Bansal)

- Completed >50 hours of testing of SiC/SiC combustor liners in the CE-9 Combustor and Material Test Facility. Rig modifications, which included fuel/air nozzle sealing changes (more uniform SiC/SiC liner temperatures), a re-designed gas analysis system, and the incorporation of Glenn-developed thin film thermocouples and multi-wavelength pyrometry were evaluated and verified. Over 260 total hours of testing provided key design data. (Verrilli, Nemets, Martin, Ng)

- High Pressure Burner Rig (HPBR) support of EBC development, both bulk materials and coatings. Milestone for demonstrating capability to test small tubes completed. Concept design developed to integrate Sector Rig (CE-9) lean transition liners into HPBR test, with demonstration test planned in 2001. (Robinson)

- Fabrication stages of thin film Pt and Pt-13%Rh thermocouples were evaluated to better understand the integrity of the thin film deposits, the welds and optimization of the fabrication process. Durability testing and microstructural characterization revealed that the current deposition and weld techniques are not optimized and do not yield quality welds. (Raj, Robinson, Opila, Barrett, Humphrey)

- Phase evolution of BSAS, incorporated in EBCs, was monitored by Raman microscopy. Conversion from the hexagonal to the desired monoclinic celsian phase was shown to occur preferentially at stress-relaxed locations. Surprisingly slow conversion of physically constrained BSAS second- phase islands likely control development of stress in these coatings. (Eldridge, Lee)

- Completed initial tests on thermal conductivity and the conductivity change kinetics of several EBC/CMC systems under combined laser thermal gradient and furnace-water vapor, cyclic conditions; proposed preliminary thermal barrier design information for the EBCs. (Zhu, Lee, Miller)

- A powder delivery system, based on the concept of a dust feeder, allows the ambient plasma spraying of small particle powders (1 ~ 10 microns) is undergoing experimental check-out for the processing of higher temperature capable EBCs. (Miller, Nguyen, Lee)

- Developed and tested new low-conductivity oxide thermal barrier coatings. Up to 50% reduction in thermal conductivity has been achieved to date. (Miller, Zhu, Eldridge, G.Leissler, S.Leissler, Setlock)

- Initiated a physics-based life prediction model for advanced TBC systems; implemented pioneering experiments and preliminary simulations for determination of TBC property change kinetics and TBC crack propagation kinetics under laser thermal gradient, thermal fatigue loading conditions. (Zhu, Miller)

- Identified airfoil leading edge sintering cracking mechanisms as the high heat flux failure modes in the engine component. Established approaches to derive the component operating temperature, stress, time regime, based on the measured sintering strains of the coating systems. (Zhu, Miller, Nesbitt)

- Preliminary infrared transmission properties of thermal barrier coatings were obtained using FTIR spectroscopy. Since TBCs are strongly scattering, the transmission configuration was modified to minimize collection of undesired scattered radiation. (Eldridge, Street)

- Isothermal kinetic studies, cyclic furnace testing in 90%H_2O/10%O_2, and HPBR screening (temperatures ranging from 1200-1400^oC) of Ames Research Center's HfB_2 and ZrB_2 ultra-high temperature material systems, to assess their aero-propulsion application potential. Significant degradation has been observed. (Opila, Nguyen, Robinson)

- HPBR improvements included a re-building of the compressor, adding new test and transition hardware sections, installing a rail system for the combustor and transition modules, expanding the quartz view port capabilities, and designing/fabricating a number of specimen holder configurations to expand beyond traditional sample geometries. (Robinson, Bunyak)

Space Transportation Projects:

- Preliminary design of a Quick Access Rocket Exhaust Exposure Rig developed using commercially available H_2/O_2 burner as a prototype system. Initial site selection is Building 24, Cell 1. (Robinson)

- Oxidation of T-300 carbon fibers in hydrogen/water-vapor environments, indicate the rate-controlling oxidation mechanism differs; chemical reaction-controlled at lower temperatures and mass transport of oxidant through gaseous boundary layer-controlled at higher temperatures. Hydrogen slows the oxidation rate in the chemical reaction-controlled regime. (Opila)

Glennan Microsystems Initiative (MEMS):

- A high temperature combustion test specimen module has been developed and demonstrated in the HPBR for durability testing of research MEM devices and sensors. The design provides protection of the critical lead wire attachments that have proven to be the primary place of failure in earlier studies. (Robinson)

Reimbursable Research:

- Successful demonstration of thermal gradient testing in the HPBR was completed for Seimens-Westinghouse proprietary materials and has led to a Space Act Agreement (SAA) for continued durability testing. (Robinson, Bunyak)

- Computational thermodynamic modeling and high temperature compatibility behavior research studies of phase-change materials provided the technical data for brake system energy storage material down selection in an Air Force funded technology demonstration effort, "Phase-Change Enhancement of Carbon-Carbon Brakes." Participants included Advanced Braking Systems Corporation and Applied Sciences, via a NASA SAA with OAI. The technology will enable cooler brake rotors, stators and surrounding wheel assembly components. (Opila)

- Wear and diagnostic studies, to aid Biomec, Cleveland, OH (GRC-GliTec SAA), of diamond-like carbon (DLC) coated and un-coated polycarbonate, enabled the assessment of DLC coating for a blood pump application. (Miyoshi, Pobuda, Dixon)

- Evaluation, of selected bonded molybdenum disulphide (MoS_2), magnetron-sputtered MoS_2, ion-plated silver, ion-plated lead, magnetron-sputtered WC/C, and plasma-assisted chemical vapor deposited diamond-like coatings enabled the down-select of the solid lubricant coating for space bearing applications, in conjunction with K Systems Corp (KSC), Beavercreek, OH (GRC-KSC SAA). (Miyoshi, Pobuda, Dixon)

- Significant portions of the overall recession observed in C/SiC by Rocketdyne/Thiokol RS-68 Ablative Nozzle Team, based on Si-O-H gaseous species formation calculations conducted for ten application environment conditions, was shown to be due to chemical interaction. GRC-GliTec SAA. (Opila)

- Developed optimized low-pressure plasma spray parameters to produce spherical rhenium metal powders for consolidation into metallurgical parts, of interest to Rhenium Alloys, Inc. Conducted via GRC-GliTec SAA. (S. Leissler)

Awards and Recognition:

- The paper "SiC Recession Caused by SiO_2 Scale Volatility Under Combustion Conditions: Part I, Experimental Results and Empirical Model" and "Part II, Thermodynamics and Gaseous-Diffusion Model" was peer-selected as the Materials Division Paper of the Year 2000. (Journal of the American Ceramic Society, 82(7), 1817-1834, 1999). (Opila, Smialek, Robinson, Fox, Jacobson)

- The paper "On the Oxidative Degradation of Nadic End-Capped Polyimides. 3. Synthesis and Characterization of Model Compounds for End-Cap Degradation Products," published in Macromolecules, 32(17), 5532-5538, 1999, was recognized for technical excellence. (M.A.B. Meador, Johnston, Frimer (SFF) and Gilinsky-Sharon (student))

- Jacobson was selected as a Fellow of the American Ceramic Society

- Conference Organizers - Jacobson chaired and organized the prestigious Gordon Research Conference on High Temperature Materials, Processes and Diagnostics 2000; Raj organized an international conference “Rate Processes in Plastic Deformation II: Towards a Unified Theory of Deformation” in October 2000, St. Louis, MO; Fox organized the closed session of the Cocoa Beach Conference in January 2000; Opila organized a session at the 198th Meeting of the Electrochemical Society in September 2000.

- Invited keynote lectures given at the International Conference on High Temperature Coatings, les Embiez, France. (Smialek, Pint) and the International Tribology Conference Nagasaki 2000 and visiting scholar at the Toyohashi University of Technology. (Miyoshi)

- Invited special issues, “Environmental Durability of Diamondlike and Diamond Films and Coatings,” in the Journal of New Diamond and Frontier Carbon Technology and “Vacuum Tribology” in the Journal of Tribology International. (Miyoshi)

Analytical Science Group FY'00 Highlights

Over 4387 Samples Analyzed in FY'00 (FY'99 4744)

CHEM	X-RAY	METLAB	SEM	PROBE	TEM
573 (803)	628 (880)	1853 (1583)	748 (849)	32 (28)	553 (570)

ASG Operations Survey provided valuable feedback, ratings were 4+ out of 5 for 75% of the ratings. Thank you for responses.

- Development of microwave dissolution technique of CuCrNb. (Nawalaniec/West/Ellis)
- Dissolution and analysis of bentonite clay samples for Zero CO_2. (Nawalaniec/Campbell)
- Direct XRF analysis of ZrO_2/Y_2O_3 TBC's as sintered disks and coatings on metal substrates.
  - The yttria stabilized zirconia was doped with rare earth oxides and analyzed intact instead of grinding and fusing into glass beads with lithium tetraborate. The coatings were created by plasma spray and electron beam physical vapor deposition techniques. (Halloran/Nesbitt)
- Analysis of Si from condensed hydroxide vapors to understand degradation of Si based ceramics.
  - The thermodynamics of the reaction of SiO2 with water vapor (from combustion) was studied. UV-Vis and ICP techniques were used to analyze the Si hydroxide species from the inside of a Pt-Rh condenser tube. (Johnson/Copeland/Myers)
- Developed a reliable EMPA calibration curve to determine dissolved oxygen content in titanium aluminides. Nine standards with various Ti/Al ratios and oxygen content (0-3 wt. % confirmed by bulk chemistry) were used to create the calibration curve. Ti/Al alloys form surface oxide scales which compromise surface analyses techniques. Using best-fit calibration curves, the influence of this oxide layer is factored into the analyses. Probe For Windows® developers have requested these results be used as the demonstration in their software documentation. (Nawalaniec/Smith/Copeland)
- TEM tripod prep and microstructure study of mutilayer thin film (Pt/TaSi2/Ti) on single crystal SiC. (Chen/Okojie)
- TEM imaging, EDS, and GIF mapping to study the interface between insitu-BN (150 nm) and CVI-BN (~400 nm) in SiC/SiC composite and grain size of UBE fiber (Chen/Yun/Bhatt).
- SRZ optimization study in advanced turbine blades via SEM/TEM. (Garg/Locci/MacKay/Ritzert)
- Deformation mechanisms in advanced turbine disk via TEM. (Garg/Ellis/Gabb)

New Equipment;

- Hitachi S-4700 automated stage
- Magnifire Optical CCD Camera/Gateway Laptop
- Struers Abrapol, Discotom 5

Laser High Cycle Thermal Fatigue Testing of Pulse Detonation Engine Combustor Materials

Dr. Dongming Zhu, Dr. Dennis S. Fox, Dr. Robert A. Miller

Pulse detonation engines (PDEs) have received increasing attention for future aerospace propulsion applications. Because the PDE is designed for a high frequency, intermittent detonation combustion process, extremely high gas temperature and pressure can be realized under the nearly constant-volume combustion environment. The PDEs can potentially achieve higher thermodynamic cycle efficiency and thrust density as compared to traditional constant-pressure combustion gas turbine engines. However, the development of these engines requires robust design of the engine components that are capable of enduring harsh detonation environments. In particular, the detonation combustor chamber, which is designed to sustain and confine the detonation combustion process, will experience high pressure and temperature pulses with very short duration. Therefore, it is of great importance to evaluate PDE combustor materials and components under simulated engine temperature and stress conditions in the laboratory.

In this study, a high cycle thermal fatigue test rig was established at NASA Glenn Research Center using a 1.5 kW CO_2 laser. The high power laser, operating in the pulsed mode, can be controlled at various pulse energy levels and waveform distributions. The enhanced laser pulses can be used to mimic the time-dependent temperature and pressure waves encountered in a pulsed detonation engine. Under the enhanced laser pulse condition, a maximum 7.5 kW peak power with a duration of approximately 0.1-0.2 ms (a spike) can be achieved, followed by a plateau region that has about 1/5 of the maximum power level with several ms duration. The laser thermal fatigue rig has also been developed to adopt flat and rotating tubular specimen configurations for the simulated engine tests. More sophisticated laser optic systems can be used to simulate the spatial distributions of the temperature and shock waves in the engine.

Pulse laser high cycle thermal fatigue behavior has been investigated on a flat Haynes 188 alloy specimen, under the test condition of 30 Hz cycle frequency (33ms pulse period, and 10 ms pulse width including 0.2 ms pulse spike). Temperature distributions were calculated using one-dimensional finite difference models. The numerical calculations show that that the 0.2 ms pulse spike can cause an additional 40ºC temperature fluctuation with an interaction depth of 0.08 mm near the specimen surface region. This temperature swing will be superimposed onto the temperature swing of 80ºC that is induced by the 10 ms laser pulse near the 0.53 mm deep surface interaction region.

Specimen failure modes were also studied after the laser thermal fatigue testing. Extensive surface cracking with the crack depths of approximately 30 µm was observed on the flat and tubular specimens tested under enhanced laser pulses and thermal cycling. The laser induced severe high frequency thermal cycles are detrimental to the potential PDE combustor superalloy materials. Preliminary results suggest that surface oxidation, alloy inclusions, grain boundaries and surface roughness have accelerated crack initiation and propagation under the intense laser pulse cyclic loads.

ALLOY DESIGN WORKBENCH 1.0

Dr. Guillermo Bozzolo, Dr. Ronald Noebe, Dr. Phillip Abel

As a result of a multidisciplinary effort involving solid state physics, quantum mechanics, and materials and surface science, the first version of a software package dedicated to the atomistic analysis of multicomponent systems was recently completed. Based on the BFS method for the calculation of alloy and surface energetics, this package includes modules devoted to the analysis of many essential features that characterize any given alloy or surface system, including 1. surface structure analysis, 2. surface segregation, 3. surface alloying, 4. bulk crystalline material properties and atomic defect structures, and 5. thermal processes, that allow us to perform phase diagram calculations. All the modules of this Alloy Design Workbench are designed to run in PC and workstation environments, and their operation and performance are substantially linked to the needs of the user and the specific application.

In response to the diverse needs of the scientific community, this tool, with the BFS method at its core, provides a simple and straightforward approach for gaining understanding of the properties of materials based on increased knowledge of system behavior at the atomic level. In what constitutes a substantial improvement over alternative quantum approximate methods with comparable computational simplicity, the BFS method for alloys is free from most of the constraints on other techniques, such as limitations in the number and type of atomic species that can be accomodated or an inability to deal with arbitrary crystal structures. This results in a general, transferable, and accurate tool for examining the characteristics of multicomponent systems.

In the last few years, the BFS method for alloys, developed at NASA Glenn Research Center (GRC), has been tested and applied to a large number of fundamental problems with consistent success. Due to the strong foundation of its general formulation, the simplicity of its implementation and its computational economy, the method has proven to be a powerful and useful tool to aid in the process of alloy design and analysis. While there are no limitations on the number of elements involved in the calculations, the crystal structure, or the input compositions, the current version of ADW is parameterized and capable of performing calculations with 15 elements in bcc form, 12 in fcc form, and 2 in the diamond structure, with this database constantly being updated.

The initial version of the Alloy Design Workbench is currently available at NASA GRC for internal use by the Computational Materials Group (CMG), a joint effort with the Ohio Aerospace Institute. ADW represents a major milestone in the ongoing computational materials program sponsored by the HOTPC program. It is envisioned that interaction with potential collaborators will help us expand the scope and capability of this tool. Eventually, such collaborations and interactions with individuals and institutions will be instrumental in leading to a generally available software package.

The BFS method is extremely economical in the input needed, which is completely provided by first-principles calculations (linearized augmented plane wave method) without reference to any experimental data. Moreover, the small number of parameters needed (three for each single element, two for each element pair), together with the universal nature of these parameters (i.e., applicable, without adjustment, to any problem dealing with these elements in a given symmetry, even in the presence of additional elements), make the BFS-based predictions surprisingly general given the limited input and the severe transferability constraints imposed on the parameters.

The package includes tools for developing large scale atomistic simulations using Monte Carlo methods, as well as tools for performing analytical calculations based on user-defined catalogues of specific atomic configurations, which provide insight into the basic features of a given system on an atom-by-atom basis.

The following type of information that been determined using ADW 1.0 on an example binary system such as Cu-Pd. 1) Analysis of bulk and surface properties of pure elements, including surface energies, surface structure, reconstruction and multilayer relaxation. 2) The formation of surface alloys for any level of coverage, thin film structure and growth patterns, and interdiffusion into pure crystals. 3) Identification of the segregating species and determination of the driving mechanisms for segregation, surface segregation profiles and their dependence on temperature, concentration and alloy crystal face, and alloy surface energies and structure. 4) Determination of bulk alloy crystal structure and properties including, phase fields, order-disorder transitions and determination of other critical temperatures. 5) Determination of the energetics of point and other atomic defects. 6) Prediction and analysis of metastable structures, 7) Determination of physical properties such as lattice parameter, compressibility, and cohesive energy as a function of composition and temperature. 8) The role and behavior of ternary and higher order alloying additions, their site preference behavior, the formation of other phases, and the partitioning of the alloying additions to these phases.

In summary, the first version of Alloy Design Workbench, a software package dedicated to the atomistic analysis of multicomponent systems, has recently been completed in response to the diverse needs of the scientific and engineering community.

THERMODYNAMICS OF TITANIUM-ALUMINUM OXYGEN ALLOYS

Dr. Evan Copeland and Dr. Nathan Jacobson

Titanium-aluminum alloys are promising intermediate temperature alloys for possible compressor applications. These materials are based on the α_2-Ti_3Al + γ-TiAl phases. The major issue with these materials is the effect of oxygen. Large amounts of oxygen dissolve in α_2-Ti_3Al. Oxidation may lead to mixed TiO_2-Al_2O_3 scales. This is not expected as phase diagram studies indicate α_2-Ti_3Al is in equilibrium with Al_2O_3. In order to understand these issues the thermodynamics of titanium-aluminum-oxygen alloys are examined.

A series of alloys near the α_2 Ti_3Al phase field of varying oxygen content were prepared. The study involves (a) determining the precise phase and composition of each alloy at temperature and (b) determining the thermodynamic activities of titanium, aluminum, and oxygen for each alloy. Compositional and phase analysis was done via standard chemical analysis, x-ray diffraction and microprobe techniques.

Thermodynamic measurements were conducted with a vapor pressure technique, using a unique double cell system, designed and fabricated at NASA Glenn. The Knudsen cell technique has been used for many years to give precise vapor pressures. In this technique vapor pressures of a particular component in an alloy and those in a pure material are measured. The ratio of two vapor pressures is the thermodynamic activity.

In order to obtain accurate measurements, a number of critical issues must be addressed. Precise measurement and uniformity of temperature in the cells is essential. Temperatures were measured with thermocouples touching the sides of the cells. Mixing of the molecular beams from each cell proved to be a major issue, so alternate materials were used, with appropriate corrections for the cross sections. Copper was used in place of aluminum; nickel was used in place of titanium.

In a mass spectrometer, vapor pressures, P, are related to intensity, I, by:

$$P = \frac{kIT}{\sigma}$$

Here k is the machine constant and σ is the ionization cross section. Initially, a second law heat was measured with the copper or nickel standard. Agreement with the tabulated values indicated that the temperature calibrations and ion intensity measurement system were functioning properly. From each standard data point, the quantity k/σ_{Cu} or k/σ_{Ni} was determined. The cross section ratios σ_{Cu}/σ_{Al} and σ_{Ni}/σ_{Ti} were obtained from previous measurements of pure copper/pure aluminum and pure nickel/pure titanium. The standard provided a check of the system and an *in-situ* value of k/σ_{Al} and k/σ_{Ti} that could then be used to calculate the vapor pressure of Al or Ti over the alloy.

Oxygen activities were measured from the $Al_2O(g) = \frac{1}{2} O_2(g) + \underline{Al}$(in alloy) equilibria (3). The equilibrium constant for this reaction is well known (4). The values for $P(Al_2O)$ and the activity of Al are measured and hence $P(O_2)$ could be determined. Oxygen partial pressures in the range of 10^{-30} can be reliably measured.

Initial measurements have been completed in the β-Ti + α_2-Ti_3Al and α_2-Ti_3Al + γ-TiAl two-phase fields. In the β-Ti + α_2-Ti_3Al two-phase field, oxygen appears to decrease the titanium activity and increase the aluminum activity. Structurally this suggests that oxygen atoms are in interstitial sites surrounded by Ti atoms. In the α_2-Ti_3Al + γ-TiAl two-phase field, the activities of Al and Ti appear less dependent on oxygen. Evidence also suggests that a large amount of dissolved oxygen in α_2-Ti_3Al is necessary for alumina formation. Further work is underway on additional alloys in this system.

Remote, Non-Contact Strain Sensing by Laser Diffraction

Marc R. Freedman

A system was developed at Glenn Research Center (GRC) for the purpose of continually monitoring, in real time, the in-plane strain tensor in opaque solids during high temperature, long-term mechanical testing. The simple, non-contacting, strain sensing methodology should also be suitable for measurement in hostile environments. This procedure has obvious advantages over traditional, mechanical, contacting techniques and is easier to interpret than moiré and speckle interferometric approaches.

A two-dimensional (2D) metallic grid of micron dimensions is applied to the metallographically prepared gage section on the surface of a tensile test specimen by a standard photolithographic process. The grid on the fixtured specimen is interrogated by a He-Ne laser and the resulting diffraction pattern is projected backwards onto a translucent screen. A charge coupled device (CCD) camera is used to image the first order diffraction peaks from the translucent screen. When the specimen is heated in a furnace, changes to the diffraction pattern can be detected. From the location of the new diffraction peaks when compared with the initial image at the same point on the grid, all four components of the in-plane deformation tensor: longitudinal, transverse and shear strain, and rigid body rotation can be calculated. In this way, bi-directional thermal expansion coefficients can be calculated. Subsequent application of load to the specimen at high temperature results in additional changes to the diffraction pattern. These changes are recorded and used to calculate bi-directional strain as a function of time (i.e., creep). Continuous translation of the laser to discrete spots covering the entire area of the grid on the specimen during the test period yields a real-time "map" of localized 2D strain over time.

This method was developed to measure strain in relatively small, flat test specimens under controlled conditions. Modifications to the current methodology, including miniaturization have been considered to encompass strain sensing of complex and/or curved surfaces. Further development would be required to provide *in-situ* strain monitoring of aero-surfaces during manufacturing and use.

GRCop-84 Development for Rocket Engines

David L. Ellis, Hee Man Yun, Bradley Lerch,Dennis Keller, Richard Holmes

GRCop-84 (Cu-8 at.% Cr-4 at.% Nb) has been developed for use in regeneratively cooled rocket engines. It possesses an excellent combination of conductivity, thermal expansion, strength, creep resistance, ductility and low cycle fatigue (LCF) life. The significantly greater properties of GRCop-84 have the potential for significant gains in engine performance and reliability compared to the currently used alloy NARloy-Z (Cu-3 wt. % Ag-0.5 wt.% Zr).

To safely use GRCop-84 in a rocket engine, it is critical to develop a detailed database of the important thermophysical and mechanical properties. Work has focused on five major properties: thermal conductivity, thermal expansion, tensile strength, creep resistance, and LCF life. The analysis of the data went beyond simply measuring the data and reporting averages. Detailed statistical analysis was conducted that allowed regression of the data over the entire temperature range tested and establishment of design minimums. The design values were expressed as simple mathematical formulas that are highly amenable to use in computer code such as Finite Element Analysis (FEA) and related computer modeling work.

During 2000, work has been completed for all five properties. The thermal expansion of GRCop-84 was found to be at least 7% lower than NARloy-Z. Lowering the thermal expansion lowers thermal stresses and increases liner life. Thermal conductivity of GRCop-84 at 70% to 83% the conductivity of copper is slightly lower than NARloy-Z but much higher than most alloys with similar strengths.

The yield strength of GRCop-84 is approximately twice that of NARloy-Z over the temperature range tested. It also retains more of its strength following simulated brazing than NARloy-Z. The higher temperatures experienced during Hot Isostatic Pressing (HIPing) somewhat degrade the properties, but HIPed GRCop-84 still retains significant advantages over NARloy-Z. The modulus of GRCop-84 is lower than pure copper. Lowering the modulus lowers the thermal stresses and again increases life.

Creep and LCF lives are much greater than NARloy-Z. Subjecting the material to a simulated braze cycle at 935ºC (1715ºF) did not adversely affect the LCF lives.

The ultimate test is to actually make a liner and test it in a rocket engine. Working with NASA Marshall Space Flight Center, two liners approximately 5.1 cm ID X 15.2 cm long (2” I.D. X 6” long) were fabricated at MSFC using Vacuum Plasma Spraying (VPS) (3) and tested at NASA GRC. Twenty-seven tests accumulating 482 seconds of hot fire testing using fuel ratios up to 7:1 were conducted. The liners showed no signs of degradation following testing.

In addition to extrusion, HIPing and VPS, GRCop-84 has been shown to be highly workable at low to moderate temperatures (250ºC to 350ºC). Sheet product approximately 22.9 cm x 50.8 cm (9” x 20”) has been rolled to a thickness of 0.1 cm (0.040”).

High Flow PMR-Polymide Composites Have Mechanical Properties Comparable to Other High Temperature Systems

Michael A. Meador

PMR polyimides, in particular PMR-15, are well known for their excellent high temperature stability and performance, and solvent resistance. However, processing of these materials is limited, for the most part, to prepreg based methods, such as compression or autoclave processing. These methods involve substantial amounts of hand labor and, as a result, manufacturing costs for components made from PMR polyimides can be high. In cost sensitive applications, these high manufacturing costs can make the use of PMR polyimide based components cost prohibitive.

Lower cost manufacturing methods, such as Resin Transfer Molding and Resin Film Infusion, have been demonstrated to reduce manufacturing costs by as much as 50% over prepreg based methods. However, these processes are only amenable to materials with melt viscosities below 30 Poise. Most PMR polyimides have melt viscosities on the order of 100 Poise or higher. Recent efforts at GRC have focused on chemical modifications to PMR polyimides to reduce their melt viscosity to the point where they could be processed by these low cost manufacturing methods without adversely affecting their high temperature properties and performance.

These efforts have led to a new family of PMR polyimides that have melt viscosities significantly lower than that of PMR-15. Reductions in melt viscosity are brought about through the introduction of molecular twists in the polymer backbone. Carbon fiber (T650-35) composites were prepared from one of these polyimides, designated PMR-Flex, by compression molding. The properties of these composites are and compared with comparable composites made from PMR-15, and PETI-RTM, a new low melt viscosity polyimide.

PMR-Flex had the lowest minumum melt viscosity of the three resins. Room temperature properties (flexural strength and modulus, short beam shear) of the PMR-Flex composites were comparable to those of the PMR-15 composites and slightly higher than those of the PETI-RTM based system (possibly due to the lower number of plies in the later). PMR-Flex composites were also tested at 232ºC and compared to comparable properties of PMR-15 laminates at 288ºC. The lower test temperatures for the PMR-Flex laminates were chosen because of the lower glass transition temperature of those laminates relative to that of the PMR-15 composites. Flexural and short beam shear strengths of PMR-Flex laminates were slightly lower that those of the PMR-15 composites. Efforts are underway to further modify this chemistry to increase the glass transition temperature and elevated temperature properties of these materials.

Long Term Durability of a Matrix for High Temperature Composites

Kenneth J. Bowles

Polymer matrix composites (PMC's) are being increasingly used in applications where they are exposed for long durations to harsh environments such as elevated temperatures, moisture, oils and solvents and thermal cycling. The exposure to these environments leads to the degradation of structures made from these materials. This also affects the useful lifetimes of these structures. Some of the more prominent aerospace applications of polymer matrix composites include engine supports and cowlings, reusable launch vehicle parts, radomes, thrust-vectoring flaps and thermal insulation of rocket motors. This demand has hassled to efforts for the development of light weight, high strength and modulus materials which have upper use temperatures over 316 ^{O}C.

A cooperative program involving two grants to the Massachusetts Institute ofTechnology and in-house work at the NASA Glenn Research Center was conducted to address the need for identifying the mechanisms and the measurement of mechanical and physical properties that are necessary to formulate a mechanism-based model for predicting the lifetime of high temperature polymer matrix composites. The polymer that was studied was PMR-15 polyimide, a leading matrix resin for use in high temperature resistant aerospace composite structures such as propulsion systems. The temperature range that was studied was from 125 to 316 ^{O}C.

The diffusion behavior of PMR-15 neat resin was characterized and modeled. Thermogravimetric analysis (TGA) was also conducted in nitrogen, oxygen and air to provide quantitative information on thermal and oxidative degradation reactions. A new low-cost technique was developed to collect chemical degradation data for isothermal tests lasting up to 4000 hours in duration. In the temperature range studied, results indicate complex behavior that was not observed by previous TGA tests, including the presence of weight-gain reactions. These were found to be significant in the initial periods of aging from 125 to 225 ^{O}C. Two types of weight loss reactions dominated at aging temperatures above 225 ^{O}C. One was concentrated at the surface of the polymer and was very active at temperatures above 225 ^{O}C. The second was observed to dominate in the latter stages of the aging at temperatures below 260 ^{O}C. This three-reaction model satisfactorily explains past findings that the degradation mechanism of PMR-15 appears to change around 316 ^{O}C. It also indicates that the second weight gain mechanism is a significant factor at temperatures below 204 ^{O}C. Based upon these results, a predictive model was developed for the thermal degradation of PMR-15 at 316 ^{O}C.

Tests were also conducted at NASA Glenn to further elucidate the theory of a three-mechanism degradation model. PMR-15 specimens were aged in air at 204, 260, 288 and 316 ^{O}C for times as long as 4400 hours. Specimens of four different Volume/Surface area were studied to evaluat geometrical effects. Weight loss and dimensional changes were measured. Three length, four width and ten thickness measurements were made for each specimen. The surface layer thickness was measured at predetermined intervals. The study confirmed that a three-mechanism degradation model is appropriate. Also, it indicates that other mechanisms may be operating. The weight loss was shared by both the oxidizing surface and the thermally degrading central core material. The ratios were dependant on the test temperature. The surface loss diminished as the temperature was reduced, and the core loss remained about the same. Dimensional shrinkage partitioning between the surface and the bulk polymer followed the same course.

Thermo-oxidative aging produces a nonuniform degradation state in PMR-15. A surface layer, usually attributed to oxidative degradation, forms. This surface layer has different properties than the central core material. A set of materials tests were designed to separate the properties of the oxidized surface layer from the properties of the inner material. Test specimens were aged at 316 ^{O}C in either air or nitrogen for up to 800 hours. The thickness of the oxidized layer, the dimensional shrinkage and the coefficient of thermal expansion (CTE) were measured directly. The nitrogen-aged specimens were assumed to be representative of thermally aged PMR-15 and therefore have the same properties as the interior material in the air-aged specimens. Four point bend tests were performed to measure the flexural modulus of both the surface layer and the inner material. Innovative bimetallic strip specimens were machined from thick aged specimens and tested to determine surface layer shrinkage and CTE. Results show that the surface layer is under tension at the cure temperature and under compressive loading at room temperature. This confirms a previous report that showed that surface cracking of PMR-15 occurs while at elevated temperature and is not due to thermal cycling during specimen weighing operations. These physical properties are of valuable use in modeling the durability and lifetime of PMR-15 structural elements because they are not readily found in the literature.

The data gained from these studies present a new understanding of the degradation of all polyimide type composite matrices. The results from this cooperative effort are of significant importance to the development of new composite materials for advanced aerospace propulsion systems.

PMR Extended Shelf Life Technology Wins 2000 R&D-100 Award

William B. Alston & Michael A. Meador

A GRC developed approach to extending the shelf-life of PMR polyimide solutions and prepregs was a recipient of this year's R&D-100 Award.

PMR polyimides, in particular PMR-15, have become attractive materials for a variety of aerospace applications due to their outstanding high temperature stability and performance. Use of these materials in components seeing temperatures of 290ºC or less can lead to substantial reductions in component and systems weight, as much as 30% over metal components. PMR-15 composites are used widely in aerospace applications ranging from ducts and external components in aircraft engines to an engine access door for the Space Shuttle Main Engine. A major barrier to more widespread use of these materials is high component costs. Recent efforts at NASA Glenn have focused on addressing the various factors that contribute to these costs in an attempt to more fully utilize these lightweight, high temperature materials.

During storage, standard PMR polyimide solutions and prepregs undergo chemical reactions that lead to the buildup of higher molecular weight species. These chemical changes affect processability and can lead to higher part rejection and scrap rates and, ultimately, higher component costs. The stability of PMR prepregs and solutions can be improved by refrigeration. However, this adds to shipping and storage costs which can be reflected in higher manufacturing costs.

This new technology increases the storage life of PMR polyimide solutions by almost a factor of 50 over standard PMR resin chemistry. Use of extended shelf life PMR solutions and prepregs can enable more repeatable processability and reduce the requirements for storage and handling of these materials. Application of extended shelf life PMR solutions and prepregs can lead to reduced manufacturing costs by reducing scrap rates and unnecessary storage and handling costs.

Thermodynamics of Volatile Silicon Hydroxides

Dr. Evan Copeland, Dr. Elizabeth Opila, and Dr. Nathan Jacobson

Silicon-based ceramics are promising candidate structural materials for heat engines. The long-term stability of these materials to environmental degradation is dependent on formation and retention of a protective SiO_2 layer. It is well-known that SiO_2 forms stable volatile hydroxides at elevated temperatures. Combustion conditions, which characteristically include high velocities, high pressures and temperatures, tend to promote a continuous formation with resulting material degradation. In order to predict the degradation of silicon-based ceramics, accurate thermodynamic data on silicon hydroxides are needed.

Three volatile silicon-hydroxide species have been proposed as being significant under combustion conditions:

$$SiO_2(s) + 2\ H_2O(g) = Si(OH)_4(g) \qquad (1)$$

$$SiO_2(s) + H_2O(g) = SiO(OH)_2(g) \qquad (2)$$

$$SiO_2(s) + \tfrac{1}{2}\ H_2O(g) = SiO(OH)(g) + \tfrac{1}{4}\ O_2(g) \qquad (3)$$

Thermodynamic data on these species are limited. A transpiration apparatus was designed and constructed to measure thermodynamic quantities for these volatile hydroxides and add to the available data.

The volatile hydroxides are formed by flowing water vapor over the solid and quantitatively collecting the products downstream for analysis. Conditions must be carefully controlled for minimization of diffusion effects, precise temperature measurements, and complete collection of products. The transpiration cell was based on a design of Hashimoto. The cell was fabricated at NASA Glenn from Pt-20Rh and is illustrated in the figure. Laser welding was used to insure a leak tight system. The cell can be used with both reactive and non-reactive carrier gases at temperatures up to 1773 K.

Initial studies have focused on experiments with an argon carrier gas containing high (0.38 atm) and low (0.15 atm) $P(H_2O)$ flowing over amorphous SiO_2. The first series at high $P(H_2O)$ was conducted at temperatures between 1073 and 1723 K and according to reaction (1) gave: ΔH_r=49.8±1.8 kJ/mole and ΔS_r=-72.6±1.3 J/mole. The second series of measurements at lower $P(H_2O)$ were taken over the temperature range of 1450 to 1723 K. According to reaction (1) these results gave: ΔH_r=56.9±0.8 kJ/mole and ΔS_r=-66.9±0.5J/mole. The results show excellent agreement with recent data from Hashimoto (1) for the formation of $Si(OH)_4(g)$: ΔH_r=56.7±1.7 kJ/mole and ΔS_r=-66.2±1.0 J/mole at an average temperature of 1600 K. The difference between the two datasets suggest $SiO(OH)_2(g)$ may be forming. Further work is underway to clarify this and obtain data for $SiO(OH)_2(g)$

An Improved Method for Surface Enhancement of Metallic Materials

Timothy P. Gabb, Jack Telesman, and Peter Kantzos

Surface enhancement methods induce a layer of beneficial residual compressive stress to improve the impact (FOD) resistance and fatigue life of metallic materials. A traditional method of surface enhancement often used is shot peening, in which small steel spheres are repeatedly impinged on metallic surfaces. Shot peening is inexpensive and widely used, but the plastic deformation of 20-40% imparted by the impacts can be harmful. This plastic deformation can cause damage in the microstructure, severely limiting the ductility and durability of the material near the surface. The plastic deformation has also been shown to promote accelerated relaxation of the beneficial compressive residual stresses at elevated temperatures. Low Plasticity Burnishing (LPB) is being developed as an improved method for surface enhancement of metallic materials.

Low Plasticity Burnishing is being investigated as a rapid, inexpensive surface enhancement method under NASA SBIR contracts NAS3-98034 and NAS3-99116, with supporting characterization work at NASA. Roller burnishing has previously been employed to refine surface finish. This concept was adopted and then optimized as a means of producing a layer of compressive stress of high magnitude and depth, with minimal plastic deformation. A single pass of a smooth free rolling spherical ball under a normal force deforms the surface of the material in tension, creating a compressive layer of residual stress. The ball is supported in a fluid with sufficient pressure to lift the ball off of the surface of the retaining spherical socket. The ball is only in mechanical contact with the surface of the material being burnished, and is free to roll on the surface. This apparatus is designed to be mounted in conventional lathes and vertical mills currently used to machine parts. The process has been successfully applied to nickel-base superalloys by the team of NASA Glenn Research Center, Lambda Research and METCUT Research, supported by the NASA SBIR Phase I and II programs, the Ultrasafe program, and the Ultra Efficient Engine Technologies program.

A comparison of the residual stresses and plasticity produced by shot peening and LPB on the nickel-base alloy IN718 has been determined. The residual stress and plasticity profiles were measured using X-ray diffraction peak shift and broadening, after repeatedly electropolishing a material layer. LPB can produce deeper compressive residual stresses with much less plasticity (%cold work) than shot peening. The high cycle fatigue resistance of this alloy increases with the application of this LPB treatment. Shot peen and LPB treated high cycle fatigue specimens were exposed at 600C/10h and then tested at room temperature. LPB treated specimens had 2-5X longer lives, even when given surface scratches normal to the load axis to simulate foreign object damage. The shot peened specimens failed at the scratches with reduced fatigue lives. Crack growth specimens were notched, pre-cracked, and LPB treated. The notch was then machined away before crack growth testing at room temperature. The LPB treatment was highly effective, completely arresting crack growth into the material. This LPB process has been successfully applied to several nickel, titanium, and aluminum alloys used in aerospace gas turbine engine and airframe applications. LPB has recently been shown surprisingly effective for treating corroded airframe materials after extended service, restoring fatigue lives to the unaged levels. LPB processing is now being extended to other alloys and applications.

Upper Temperature Limit of EPM EBC Established

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Silicon-based ceramics, such as SiC/SiC composites and Si_3N_4, are the prime candidates for hot section structural components of next generation gas turbines. A key barrier to such an application is the rapid recession of silicon-based ceramics in combustions environments due to the volatilization of silica scale by water vapor. Environmental barrier coatings (EBC's) were developed to prevent the recession in the High Speed Research-Enabling Propulsion Materials (HSR-EPM) Program. An investigation at the NASA Glenn Research Center at Lewis Field was undertaken to establish the upper temperature limit of the EPM EBC, under the Ultra Efficient Engine Technology (UEET) program.

The EPM EBC consists of three layers: a silicon bond coat, a mullite ($3Al_2O_3 \bullet 2SiO_2$) or mullite-based bond coat, and a barium-strontium-aluminum silicate (BSAS: $BaO_x \bullet SrO_{1-x} \bullet Al_2O_3 \bullet 2SiO_2$) top coat. Volatility and environmental/chemical stability of the EBC in water vapor were the key criteria in establishing the upper temperature limit in this study.

The volatility was investigated by exposing monolithic hot-pressed BSAS coupons in 50% H_2O/balance O_2 at 1200 – 1500°C while continuously monitoring the weight change using a microbalance. Rapid initial weight loss is an artifact of the experimental procedure. A linear weight loss was observed, with the rate increasing with temperature. In Table I the weight loss rate is converted to recession rate in a simulated combustion environment (pressure: 6 atm; gas velocity: 25 m/sec; pH_2O: 0.6 atm). The recession rate of BSAS determined in a high-pressure burner rig at 1300°C agreed with the converted recession rate in Table I within a factor of two, supporting the validity of the conversion. The high recession rate at T > 1400°C can become an issue, especially for thin EBC's (< 100 µm).

Table I. Recession of BSAS (1000 hrs; 6 atm; gas velocity: 25 m/sec; pH_2O: 0.6 atm)

Temperature (°C)	1300	1400	1500
Recession (µm)	7.5	17.8	72

For environmental/chemical stability study, EBC-coated silicon-based ceramic coupons were exposed to 90% H_2O/balance O_2 at 1300 – 1480°C under thermal cycling. Each cycle consists of 1 hr at high temperature and 20 min at room temperature. The exposed coupons were subsequently prepared metallographically and the environmental/chemical degradation was characterized using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). Silicon was not applied on coupons exposed to 1440 and 1480°C to avoid the melting of silicon (1410°C). The EBC cross-section at 1400°C remained similar to the as-processed state, except for some limited oxidation at the silicon/mullite-based bond coat interface. In contrast, a severe reaction developed at 1440 and 1480°C: a thick scale formed at the silicon/mullite-based bond coat interface, a new layer formed on top of the BSAS (area B in Figs 2b and 2c), and an extensive reaction occurred between mullite and BSAS within the bond coat. X-ray diffraction indicates that the new top layer is a glass, which EDS analysis shows has the same composition as the interface scale. According to the $BaO\text{-}SiO_2\text{-}Al_2O_3$ phase diagram, BSAS and silica form a low melting (1296°C) silicate phase. It is therefore suggested that the glass phase is a reaction product between the silica (grown on SiC as a result of oxidation) and the BSAS. The glass moved to the EBC surface, presumably due to capillary forces or the low surface energy of the glass phase. The significant thinning of the BSAS top coat at $T > 1440^{o}C$, and the channel that developed between the interface scale and the top glass layer, supports this suggestion. The presence, and therefore the formation, of a low melting glass must be avoided because it leads to the spallation of the EBC.

Based on the foregoing results, one may conclude that the upper temperature limit of EPM EBC should be set at $1400^{o}C$ for a targeted 1000 hour life.

Environment Conscious Ceramics (Ecoceramics)

Dr. Mrityunjay Singh

Since the dawn of human civilization, there has always been a delicate balance between expanding the human frontiers and co-existing with the ecosystem. In the new millennium, in order to sustain a healthy life in harmony with nature, it will be extremely important to develop various materials, products, and processes and minimize any harmful effects on the environment. Environment conscious ceramics (Ecoceramics) are a new class of materials which can be produced with renewable resources (wood) and wood wastes (wood sawdust). Wood is one of the best and most intricate engineering materials created by nature. Natural woods of various types are available throughout the world. In addition, wood sawdusts are generated in abundant quantities by sawmills. Environment conscious ceramic materials, fabricated via the pyrolysis and infiltration of natural wood-derived performs with silicon have tailorable properties with numerous potential applications.

Silicon carbide-based ecoceramics are fabricated by reactive infiltration of wood-derived carbonaceous preforms with molten silicon or silicon-refractory metal alloys. These carbonaceous preforms are fabricated by pyrolysis of solid wood bodies up to 1000°C. The pyrolysis is carried out in a flowing nitrogen atmosphere. Melt infiltration is then carried out at temperatures above the melting point of silicon or the silicon alloy. The microstructure and mechanical properties (flexural strength and compressive strength) of a wide variety of SiC-based ecoceramics have been measured. Ecoceramics have tailorable properties and behave like ceramic materials manufactured by conventional approaches. The wood derived carbonaceous performs have been shown to be quite useful in producing porous or dense materials having different microstructures and compositions. Detailed thermomechanical characterization of a wide variety of silicon carbide-based ecoceramics is underway.

Feasibility of Actively Cooled Silicon Nitride Airfoil for Turbine Applications Demonstrated

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Nickel base superalloys currently limit gas turbine engine performance. By actively cooling, the temperature range of service of nickel base superalloys in current gas turbine engines has been extended, but the margin for further improvement appears modest. Therefore, significant advancements in materials technology are needed to raise turbine inlet temperatures (TIT) above 2400^0F to increase engine specific thrust and operating efficiency. Because of their low density, and high temperature strength and thermal conductivity, in-situ toughened silicon nitride ceramics have received a great deal of attention for cooled structures. However, high processing costs, and low impact resistance of silicon nitride ceramics have proven to be major obstacles for wide spread applications. Advanced rapid prototyping technology in combination with conventional gel casting and sintering can reduce high processing costs, and offers the possibility of an affordable manufacturing approach.

NASA Glenn researchers in cooperation with a local university and an aerospace company are developing actively cooled and functionally graded ceramic structures. The objective of this program is to develop cost effective manufacturing technology, and experimental and analytical capabilities for environmentally stable, aerodynamically efficient, foreign object damage (FOD) resistant in-situ toughened silicon nitride turbine nozzle vanes, and to test these vanes under simulated engine conditions.

Starting with computer aided design (CAD) files of an airfoil and a flat plate with internal cooling passages, the permanent and removable mold components for gel casting of ceramic slips were made by stereo-lithography and Sanders machines, respectively. The gel cast part was dried and sintered to final shape. Several in-situ toughened silicon nitride generic airfoils with internal cooling passages have been fabricated. The uncoated and thermal barrier coated (TBC) airfoils and flat plates were burner rig tested for 30 min without and with air-cooling. Without cooling, the surface temperature of the flat plate reached ~2350^0F. With cooling, the surface temperature decreased to ~1910^0F- a drop of ~440^0F. This preliminary study demonstrates that near net shape silicon nitride airfoil can be fabricated and that silicon nitride can sustain severe thermal shock and the thermal gradients induced by cooling and is thus a viable candidate for cooled components.

Creep/Rupture Behavior of Melt Infiltrated SiC/SiC Composites

Janet Hurst

The failure behavior of melt-infiltrated SiC/SiC ceramic matrix composites is under investigation as part of NASA's UEET program. This material was originally developed under the HSR Office's Enabling Propulsion Materials Program. Creep and rupture data provide accelerated testing information to predict material behavior under engine use situations (1500F to 2400F). This information gives insights into various material development paths to provide improved composites as well as understanding of failure mechanisms.

Microscopy suggests that creep and rupture of these materials can best be considered as a probabilistic property, rather than a material property. Fiber failure occurs first in isolated regions, while stronger adjacent fibers remain intact. As oxidation kinetics of SiC are well understood, oxide scales can be used as a measure of the length of time various regions of the composites have been exposed to the environment, hence providing vital information regarding the sequence of failure. An observed oxide scale is indicative of an early failure of this tow of fibers while adjacent tows remain oxide free, suggesting failure much later in time. The path of various cracks can be followed throughout the composite in this manner, suggesting failure mechanisms.

Most CMC applications require stringent dimensional tolerance. In addition, excessive amounts of creep strain have been shown to degrade material strength. Current work has demonstrated that improvements to the reinforcing fiber have been shown to dramatically improve creep behavior.

Creep and rupture testing are providing guidance for ongoing improvements to the high temperature thermomechanical behavior of SiC/SiC composites as well as fundamental understanding of the failure mechanisms involved in these materials.